Application Note

SEMICONDUCTOR MANUFACTURING SOLUTIONS

$MoCl_5$ Ampoule Depletion Detection and Leak Detection with a SemiQCM® Sensor

An INFICON SemiQCM sensor installed on the foreline of a semiconductor CVD chamber offers an effective solution for both precursor delivery fault detection and air leak detection.

Background

Detecting precursor ampoule depletion during semiconductor processing is crucial, as the industry demands unwavering precision and consistency at every stage of fabrication. Advanced deposition techniques such as chemical vapor deposition (CVD) and atomic layer deposition (ALD) rely on a stable, well-regulated supply of precursors to form thin films with exacting thicknesses and properties. The use of ampoules is a common method of storing and dispensing the precursors for CVD and ALD. If an ampoule runs dry mid-process without timely detection, it can result in film non-uniformities, defects, and device failures, jeopardizing the integrity of an entire wafer batch. Such disruptions not only degrade yield but also escalate production costs and waste valuable materials.

An INFICON SemiQCM sensor, based on quartz crystal microbalance technology, provides an effective solution for both precursor depletion detection and air leak detection in this application. This technology harnesses the converse piezoelectric effect, where an applied voltage induces oscillations in the quartz crystal. The oscillation frequency is extremely sensitive to mass variations, meaning even the slightest addition or removal of material on its surface triggers a measurable frequency shift. In this application, the quartz crystal is securely embedded within a sensor, ensuring stability and enabling highly precise frequency measurements with the IMM-200 quartz crystal frequency monitor in Fig. 1. This high sensitivity enables precise, real-time monitoring of mass changes, making SemiQCM an indispensable tool in applications requiring exceptional accuracy and stability.

Mo Film Deposition and Its Precursor ($MoCl_5$)

Molybdenum (Mo) thin films are essential in semiconductor manufacturing due to their outstanding electrical, thermal, and mechanical properties. With a high melting point, low thermal expansion coefficient, low resistivity, and excellent thermal conductivity, Mo is widely used in diffusion barriers, electrodes, photomasks, power electronic device substrates, and low-resistivity interconnects. Its exceptional stability under extreme conditions and minimal energy loss make it a preferred choice for metal electrodes in semiconductor devices. In particular, Mo thin films play a crucial role in metal-oxide-semiconductor field-effect transistor (MOSFET) gate electrodes, where precise electrical control is critical for device performance. As semiconductor technology advances, optimizing Mo film deposition for high conformality and fast deposition rates remains a key focus to support efficient mass production and ensure superior device reliability.

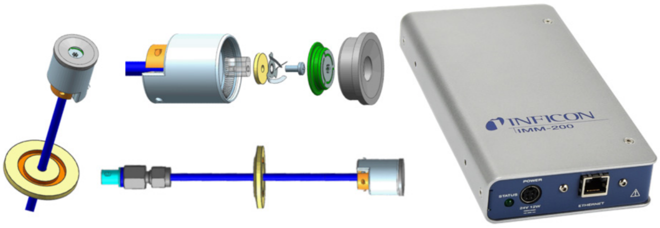

Figure 1. Schematic diagram of a SemiQCM sensor without the quartz crystal and an IMM-200 controller.

CVD is a crucial method for depositing Mo thin films in semiconductor manufacturing, offering superior conformality, step coverage, and precise thickness control. Molybdenum pentachloride ($MoCl_5$) is a highly effective precursor for CVD of Mo thin films, prized for its excellent volatility, strong reactivity, and efficient decomposition characteristics. As a solid at room temperature, $MoCl_5$ readily sublimates under controlled conditions, ensuring precise vapor-phase delivery for uniform and high-quality film growth. Its strong oxidizing nature enables rapid and efficient reactions with reducing agents like hydrogen (H_2), facilitating the deposition of high-purity Mo films with exceptional adhesion, smooth morphology, and low resistivity. Moreover, $MoCl_5$ offers excellent thermal stability, ensuring a consistent and reliable precursor supply throughout the deposition process. These attributes make it an ideal choice for advanced semiconductor applications, including diffusion barriers, gate electrodes, and interconnects, where precision, performance, and scalability are critical.

SemiQCM in Ampoule Depletion Detection

INFICON tackles the challenge of ampoule depletion detection for $MoCl_5$ by strategically positioning SemiQCM sensors along the foreline of semiconductor processing tools. The foreline is a crucial location where the temperature is lower than that of the process chamber, creating conditions that cause precursor vapors to condense on its walls and the SemiQCM sensor's crystal surface. As $MoCl_5$ accumulates, it induces measurable frequency shifts due to the added mass, providing real-time insights into precursor flow dynamics. By continuously monitoring these frequency variations, the SemiQCM system accurately detects precursor depletion, triggering alerts or automated responses to maintain process stability.

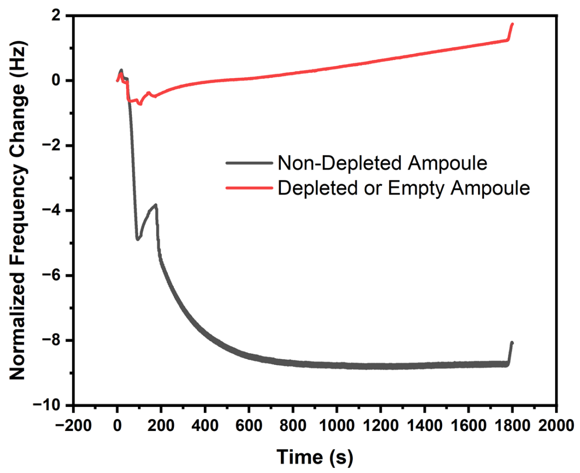

Figure 2. Plots of the frequency shift measured by a SemiQCM sensor between precursor depleted ampoule and non-depleted ampoule when operating under the same process recipe.

As shown in Fig. 2, the frequency shift from the SemiQCM clearly distinguishes between depleted and non-depleted ampoule when operating under the same process recipe. A noticeable frequency drop corresponds to the accumulation of condensed precursor on the SemiQCM sensor, indicating the presence of precursor in the system. In contrast, a stable frequency suggests that no precursor is condensing on the sensor, signifying that the ampoule is depleted. By continuously analyzing these frequency variations in real time, the system provides a highly reliable indication of precursor exhaustion, enabling timely ampoule replacement.

SemiQCM in Leak Detection

By monitoring SemiQCM frequency variations, the SemiQCM sensor can not only detect the depletion of the precursor ampoule but also identify potential leaks within the chamber or processing system, ensuring enhanced process stability and reliability. When the precursor is not yet depleted, the SemiQCM sensor exhibits a distinctly different frequency shift in the presence of a leak compared to a leak-free system. As illustrated in Fig. 3, an air leak was detected along the pathway between the precursor source and the chamber, leading to a significant and rapid frequency response from the SemiQCM sensor. This strong deviation indicates abnormal precursor behavior, allowing for early leak detection and preventing potential process disruptions.

Under leak conditions, the SemiQCM sensor exhibits a more significant frequency shift due to oxygen (O_2) intrusion into the carrier gas line, which reduces the MoCl$_5$ partial pressure and disrupts deposition. While the wafer experiences severe thickness loss due to precursor starvation, any MoCl$_5$ or Mo on the SemiQCM sensor reacts with O_2, forming a high-density Mo/O compound. This oxidation drastically increases the detected mass, causing a much larger frequency drop compared to normal conditions. The combined benefits of precursor depletion monitoring and oxidation-induced mass accumulation make SemiQCM a highly sensitive tool for detecting system leaks.

Summary

SemiQCM based on quartz crystal microbalance technology enables precise monitoring of MoCl$_5$ depletion and in situ air leak detection in semiconductor processing by analyzing frequency shifts. For MoCl$_5$ depletion detection, SemiQCM frequency decreases as MoCl$_5$ condenses on the sensor, indicating a sufficient precursor supply. When the ampoule is depleted, condensation stops, stabilizing the frequency, allowing timely ampoule replacement to maintain process continuity. For air leak detection, oxygen (O_2) intrusion into the carrier stream lowers MoCl$_5$ partial pressure, reducing wafer thickness while oxidizing MoCl$_5$ or Mo on the SemiQCM sensor. This oxidation significantly increases mass, causing a sharp frequency drop, effectively signaling a system leak. By leveraging the benefits provided by a SemiQCM sensor, semiconductor manufacturers can ensure real-time process stability, minimize defects, and optimize production efficiency.

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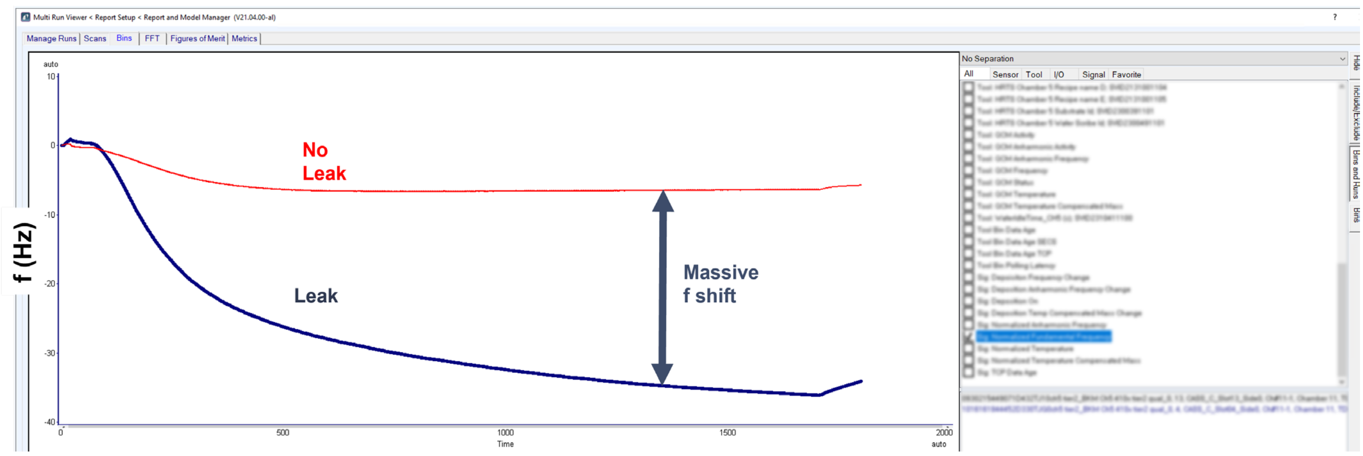

Figure 3. Normalized frequency shift measured by a SemiQCM sensor under conditions where the precursor is not depleted, comparing the presence and absence of an air leak event.

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